

# PATENT SPECIFICATION

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## DRAWINGS ATTACHED

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## (54) PRODUCTION OF ALUMINA PELLETS

(71) We, ALUMINUM COMPANY OF AMERICA, a Corporation organized and existing under the laws of the State of Pennsylvania, United States of America, of Alcoa Building, Pittsburgh, State of Pennsylvania, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to production of alumina of high sorptive capacity. More particularly, it relates to increasing surface area and adsorption capability of pelletized alumina.

Water pelletizing of activated alumina, while producing strongly bonded pellets, may sometimes have a detrimental effect on the sorptive capacity of the finished pelletized product because of the exothermic nature of the reaction resulting in an undesirably high temperature in the aging step which conventionally follows. Due partly to its lack of susceptibility by itself to water pelletizing, it has heretofore been unknown how to use alumina hydrate which has not been activated to produce a strong alumina end product of good sorptive capacity. Therefore, development of a process which employs both activated and unactivated alumina for producing a high surface area alumina of good sorptive capacity without the process being adversely affected such that the strength of bonding or the sorptive capacity of the product becomes too low to satisfy the high surface area and high sorptive capacity and other requirements for commercial use as a desiccant represents a highly desirable result.

We have now developed a method of improving the sorptive capacity of pelletized alumina and producing an improved desiccant-grade alumina which has a higher sorptive capacity than that of prior art water-pelletized activated alumina.

In accordance with the present invention there is provided a process for producing alumina pellets having a high surface area and high sorptive capacity which comprises pelletizing a mixture of activated alumina and alumina hydrate in the presence of water and aging the resulting product. The preferred activated alumina used in the invention is porous, sorptive alumina produced by heating alumina hydrate to a loss on ignition (LOI) of 3 to 14% by weight. While the activated alumina may be prepared by conventional procedures, use of so-called "flash activation" insures best results in practising the invention. In flash activation, particulate alumina hydrate is fed into an upwardly-flowing stream of hot gas in a furnace, in which the particles are entrained and heated in a few seconds to a temperature sufficient to drive off combined water therefrom, and the resulting particles are separated from the gas stream by a cyclone separator at the top of the furnace. Preferably the activated alumina has an LOI of 4—7%, a surface area of 220—260 m<sup>2</sup>/g, and a sorptive capacity of 17—19% at 60% relative humidity (RH).

In a preferred embodiment of the present invention Bayer alumina hydrate is used both as the source of activated alumina and as the alumina hydrate used in the mixture to be pelletized.

In the pelletizing operation a mixture of activated alumina particles, alumina hydrate particles and water is compacted together to form a unitary body, for example, by pressing the mixture or by tumbling the alumina hydrate and activated alumina particles together while spraying water thereon.

The median particle size of the feed mixture for water-pelletizing is preferably from 10 to 18 microns. The heat of wetting of the mixture of activated alumina and alumina hydrate is at least 10 cal/g. The activated alumina and the alumina hydrate may be ground to the desired fineness either before

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or after mixing. When a conventional rotating drum pelletizer is used in water pelletizing the alumina hydrate and activated alumina, the average retention time is 5—15 minutes for production of pellets having a 0.7—0.8 g/cc loose bulk density after final activation. The resulting pellets may be screened to the desired size and then after-rolled, and are then aged at elevated temperature. For the aging step we prefer to use closed containers. For a mixture containing about 50% flash-activated hydrate (FAH), the maximum aging temperature is generally about 210°F., this temperature resulting from the exothermic nature of the pelletizing reaction, that is, from the above-mentioned preferred heat of wetting of the activated alumina/alumina hydrate of at least about 10 cal/g. Preferred aging time is 2 to 10 hours. Aging is preferably followed by drying, preferably at 200—250°F., and reactivation, preferably at low humidity and with step-wise heating to a maximum temperature of 800—850°F. When the foregoing preferred procedure is followed, the final activated product has an LOI of 2.6—3.9% and a 60% RH sorptive capacity of at least 20%, and may also have a surface area of up to about 400 m<sup>2</sup>/g or higher.

While we do not wish to be bound by any particular theory of how a product of improved sorptive capacity is achieved according to the process of our invention, it appears that by using at least some alumina hydrate along with the activated alumina, the rehydration reaction temperature is restricted, that is, the exothermic nature of the reaction is lessened. Also, there is less tendency for particles to cling to one another and form non-spherical pellets, thereby improving the pelletization operation. The presence of the alumina hydrate is believed to lessen the amount of heat evolved in the pelletizing reaction and to dissipate some of the heat evolved, thereby lowering the temperature during the aging step which preferably follows the pelletizing and improving the sorptive capacity of the product. Use of hot water during pelletization permits rehydration of the activated hydrate component to proceed more rapidly, thereby producing pellets which are of sufficient strength to be screened directly and then rolled and aged. Use of hot water in pelletizing further reduces the maximum aging temperature as a result of dissipation of heat during forming and rolling prior to aging and it is preferred to use water at a temperature of from 80° to 180°F. For example, if 155°F. water is used in pelletizing a mixture containing about 40—60% by weight alumina hydrate, the temperature which the resulting pellets reach during aging is restricted to about 150°F. and the sorptive capacity of the final product after aging thereby further increased. The material con-

taining the alumina hydrate and activated alumina which is to be pelletized according to our invention may, however, contain up to 75% by weight alumina hydrate, with the product usually having a somewhat lower strength at a level as high as 75%.

For a better understanding of our invention reference will now be made to the accompanying drawing.

In the drawing, which is in schematic flow-sheet form, activated alumina and alumina hydrate ground to a 10—18 microns median size (either separately ground before mixing or ground in admixture) are pelletized with water at 10. The pelletized product is screened at 12 and then rolled at 14 according to any standard procedure known to the art, to improve its surface characteristics. After aging at 16, it is activated at 18 to form a high-sorptive end product alumina, which may then be introduced to a product surge tank (not shown) and bagged.

The following examples are further illustrative of our invention.

**Example 1**  
1,000 lbs of 3—6 mesh (U.S. Standard Sieve) alumina hydrate pellets were formed at 3 lbs/min by water pelletizing a mixture containing 45% by weight activated Bayer alumina trihydrate of 6.3% LOI, 232 m<sup>2</sup>/g surface area and 18.4% sorptive capacity at 60% RH and 55% Bayer alumina trihydrate. The water binder temperature was 155°F. The after-rolled pellets were aged for 6 hours and then dried at 200°F. They were then activated by heating in a continuously air-purged furnace. The product averaged 3.1% LOI, 416 m<sup>2</sup>/g and 23.3% sorptive capacity at 60% RH (as determined by a standard static sorption test).

**Example 2**  
A 600-lb batch of high-sorptive alumina was prepared using substantially the same procedure as that of Example 1 except that the activated alumina used averaged 4.8% LOI, 260 m<sup>2</sup>/g and 18.5% sorptive capacity. Low-humidity, final activation gave a product averaging 3.1% LOI, 424 m<sup>2</sup>/g, and 24.2% sorptive capacity.

It is seen from the foregoing description and examples that we have provided an improved process for preparing alumina of high surface area and high sorptive capacity. According to our process the aging temperature, which is highly important in producing alumina of the desired high sorptive capacity, may be controlled by appropriate variation of the several factors which affect it, for example, the heat of wetting of the activated alumina, the percentage of activated alumina in the starting activated alumina/alumina hydrate mixture, the water binder tempera-

ture, the rate of agglomeration, and the surface area of the heat dissipation system used in the process. The heat of wetting of the activated alumina varies directly with its static sorptive capacity, which is in turn a function of the loss on ignition. Accordingly, by using the preferred low loss on ignition, high sorptive capacity activated alumina, heat of wetting values will generally fall within the range of 34—44 cal/g. The desired heat evolved and the desired aging temperature may also be obtained by adjusting the amount of activated alumina incorporated in the pelletizer feed mixture. The more alumina hydrate used in the mixture, the less heat evolved and the lower the aging temperature.

The water binder temperature may be used as a secondary control of the aging temperature. By increasing the water binder temperature a reaction may be instigated earlier, with resultant increased heat loss to surroundings such as ambient air and after roller.

Thus, less heat is evolved in the aging container which may be used in the aging step, and a reduced aging temperature is brought about.

A further advantage of the process of this invention is that the aging temperature may be kept fairly constant within the desired range while increasing the pelletizing rate by increasing the rate of heat dissipation, for example, by making the surface area of the apparatus used, such as an after roller, larger or by water cooling the apparatus to extract more heat from the system prior to the aging. The product of the present invention is especially useful as a desiccant.

**WHAT WE CLAIM IS:—**

1. A process for producing alumina pellets having a high surface area and high sorptive capacity which comprises pelletizing a mixture of activated alumina and alumina hydrate in the presence of water and aging the resulting product.
2. A process according to claim 1 wherein the activated alumina in the presence of alumina hydrate comprises a mixture of activated alumina and alumina hydrate having a heat of wetting of at least 10 cal/g. 50
3. A process according to claim 1 or 2 wherein the activated alumina used is flash-activated alumina. 55
4. A process according to claim 3, wherein the activated alumina used is flash-activated alumina of from 3 to 14% loss on ignition.
5. A process according to any of claims 1 to 4 wherein the alumina hydrate used comprises up to 75% by weight of the total amount of activated alumina and alumina hydrate. 60
6. A process according to any of claims 1 to 5, wherein the water used in the water pelletizing is at a temperature of from 80°F. to 180°F. 65
7. A process according to any of claims 1 to 6, wherein the median particle size of the alumina hydrate and activated alumina is from 10 to 18 microns. 70
8. A process according to any of claims 1 to 7 wherein the pelletized particles are screened, rolled, aged and activated by heating. 75
9. A process as claimed in claim 8 wherein in the aging is conducted at a temperature not exceeding 210°F. and the product after being activated by heating has a 60% RH sorptive capacity of at least 20%. 80
10. A process for producing alumina pellers having a high surface area and high sorptive capacity substantially as herein described in either of the Examples. 85
11. Alumina pellets having a high surface area and high sorptive capacity whenever produced according to the process of any of claims 1 to 10.

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